PB-238 474

DEVELOPMENT OF AN ACETYLENE MONITOR AT THE PART PER BILLION LEVEL

W. Dencker, et al

Beckman Instruments, Incorporated

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16. ABSTRACT

The purpose of this contract was to develop instrumentation for the collection and analysis of atmospheric acetylene at the part per billion level. The reason for measuring acetylene is that it comes primarily from vehicular exhausts and thus can be used as a tracer of pollution from urban to rural areas.

The approach used was an automated concentration technique whereby one liter of air was collected in a 1-1/2 x 1/8" tube filled with molecular sieve. The trapped contents were then automatically flushed into a gas chromatograph for separation and assay of acetylene. This collection and analysis system was designed to automatically perform three acetylene analysis per hour at acetylene concentrations ranging from .03 to 250 PPB.

A portable collection device, in addition to the automated collection and analysis instrumentation, was also developed for the collection of acetylene in locations far removed from the analyzer. The collection devices used in the portable sampler were identical to the collection device on the gas chromatograph.

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DEVELOPMENT OF AN ACETYLENE MONITOR AT THE PPB LEVEL

by

Beckman Instruments Inc.,
1630 So. State College Blvd.,
Anaheim, California 92806

Contract No. 68-02-0778
Program Element No. 1A1003
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EPA Project Officer: James D. Mulik

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National Environmental Research Center
Research Triangle Park, N.C. 27711

Prepared for

OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
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August 1974

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Publication No. EPA-650/2-74-056

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This report was submitted in fulfillment of EPA-650/2-74-056, Contract No. 68-02-0778 by Beckman Instruments, Inc., under the sponsorship of the Environmental Protection Agency. Work was completed as of July 1974.

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SECTION 1

INTRODUCTION

GENERAL

On May 30, 1973, Beckman Instruments, Inc., Advanced Technology Operations, was awarded Contract 68-02-0778 by the Environmental Protection Agency, Research Triangle Park, North Carolina. This contract was for the construction and evaluation of two analyzers to measure acetylene at the subpart-per-billion level. The analyzers were delivered on schedule within the original budget, and meet or exceed all EPA specifications. This report describes these instruments, their development, and laboratory testing.

OBJECTIVES

The acetylene content of ambient air is of interest to EPA because it offers a means of evaluating the air mass transport from urban to rural areas. Acetylene was considered for use as a tracer gas in these studies because the primary source of acetylene in the atmosphere is from the combustion of gasoline in automobile engines that predominate in urban areas, and because acetylene is photochemically unreactive.

EPA funded this development contract because there was no commercial system available that could automatically measure acetylene below the parts-per-billion level.

In the contract Work Statement EPA suggested using an automated concentrator to trap the acetylene from one liter of air followed by injection of the trapped contents into a gas chromatographic column for separation. The system gas flow should be controlled by electrically operated pneumatic valves programmed by adjustable timers. The entire analysis must be capable of repetition every 20 to 30 minutes.

The analyzers needed to be compact and easily transportable. A flame ionization detector should be used containing an amplifier with a sensitivity of 5×10^{-12} amperes full scale with a background baseline noise lower than 10^{-13} amperes.

Beckman modified a Model 6800 Air Quality Chromatograph to meet these requirements. This instrument already contained the valves, timers, flame ionization detector, and amplifiers that meet EPA's requirements.

EPA required that a suitable gas chromatograph column and trap be developed. The instrument should be programmed to automatically operate both the traps and column.

Details of the modifications made to the Model 6800 Air Quality Chromatograph to meet these requirements are described in Section IV.

The trap required by EPA must be tested to measure the following:

- trapping capacity for acetylene;
- trapping efficiency for acetylene;
- temperature required to desorb 100 percent of the acetylene;
- trapping efficiency and desorption characteristics of iso and normal butane as well as iso and normal pentane;
- effect of humidity on trapping efficiency;
- stability of trapping material both before and after collecting acetylene. A storage life of at least 3 weeks is desired.

The trap design description and rational is given in Section IV.

A device should be constructed to collect acetylene for subsequent analysis at a later date. The design, construction, and test of the Remote Sampler to meet this requirement is described in the following section.

SECTION II

CONCLUSIONS

This report describes how a Beckman Model 6800 Air Quality Chromatograph was successfully modified to automatically measure subpart-per-billion concentrations of acetylene in the atmosphere. A simple, ambient temperature trap was developed that was adapted to remote operation. Long-term unattended operation was demonstrated.

We have demonstrated the feasibility of automatically measuring not only acetylene at the subpart-per-billion level, but normal butane and normal pentane as well.

SECTION III

RECOMMENDATIONS

Beckman recommends that EPA proceed with field-testing the instrument and its Remote Sampler.

We also recommend that EPA consider using this technique for measuring very low levels of almost any compound that is feasible to run through a gas chromatograph. For example, this method may be usable to detect small concentrations of carcinogens in the air.

SECTION IV

DESCRIPTION OF THE INSTRUMENT

GENERAL

Beckman met the contractual requirements for the analyzer by modifying two Model 6800 Air Quality Chromatographs as follows:

- Modified the gas flow system
- Modified the instrument's automatic operating sequence
- Designed and developed a new sample collection device

One of the modified Model 6800 instruments is shown in Figure 1.

Three traps, valves, a sample pump, timer, and flowmeter were packaged as a Remote Sampler Unit, designed to collect samples in the field. A detailed description of the Remote Sampler is given in this section.

ACETYLENE ANALYZER

Manual Operating Controls

Table 1 lists the controls and indicators on the front panel of the Beckman Model 6800 Air Chromatograph which has been modified to perform acetylene analyses in ambient air.

Analyzer Gas Flow System

The gas flow system for the Beckman Model 6800 Air Quality Chromatograph was modified as shown in Figures 2 and 3.

Sample Collection - The valves (Figure 2) are shown in position to collect an acetylene sample on the 1/8-inch acetylene trap. Both valves are in the OFF position. The sample gas is drawn into the system by a vacuum pump. The rate of sample flow is controlled by a flow control needle valve and monitored by a visual flowmeter.

The acetylene is removed from the air sample stream by the 1/8-inch acetylene trap. (The purpose of the 1/16-inch acetylene trap is discussed in the following pages.) Note that the two acetylene traps are mounted outside the instrument case so that they can be easily removed. This allows traps from the Remote Sampler to be conveniently placed into the analyzer so that their acetylene content can be measured.

The sample inlet line goes through a 3-way solenoid valve that permits either an atmospheric sample or calibration gas to be drawn into the instrument. The sample chosen is drawn through a 7-micron-sintered filter that removes the particulates, and through a pretrap to valve V-B. The

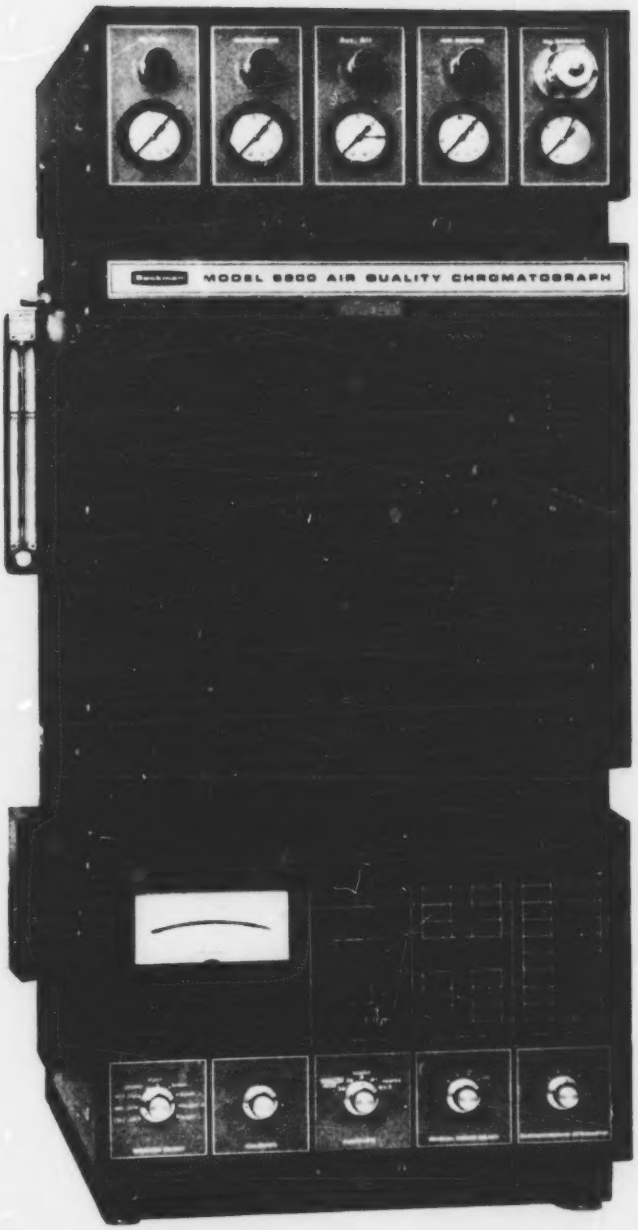

Figure 1. A Beckman Model 6800 Air Quality Chromatograph Modified to Measure Acetylene in the Atmosphere at the Subpart-Per-Billion Level

Table 1 (sheet 1 of 2). Controls and Indicators

H_2 Fuel Pressure control knob

H_2 Fuel Pressure indicator

Burner Air Pressure control knob

Burner Air Pressure indicator

Auxiliary Air Pressure control knob

Auxiliary Air Pressure indicator

Air Service Pressure control knob

Air Service Pressure indicator

H_2 Carrier Pressure control knob

H_2 Carrier Pressure indicator

Oven Heater (indicator light)

*Catalytic Heater (indicator light)

"Monitor Select" (rotary switch selecting which of the following functions read out on the panel meter)

- Cycle Time
- Amplifier Output
- Auto Zero (indicates level of zero suppression)
- Calibrate
- Memory 1
- Memory 2
- Memory 3 not used in this instrument
- Memory 4
- Memory 5

Calibrate (manual adjustable potentiometer)

"Function" (rotary switch selecting which of the following functions are performed)

- Two analyses/hour
- Three analyses/hour
- Sample (for use when analyzing samples collected by the Remote Sampler--vacuum pump does not operate with switch in this position)
- Heater 1 (turns on trap heater in manual mode)
- Heater 1-2 (turns on trap heaters in manual mode)

Manual Range Select (rotary switch selecting coarse manual attenuation ranges)

*Catalytic heater is used in the air line to remove hydrocarbons

Table 1 (sheet 2 of 2). Controls and Indicators

Chromatograph Attenuator (rotary switch selecting fine manual attenuation ranges)

Control Functions (Toggle Switches)

- Single-Continuous
- Manual-Auto
- Calibrate-Operate
- Manual Att. - Auto Att.

Manual Functions (Toggle Switches)

- Start
- Reset
- Flame Out Override
- Auto Zero
- Calib. Std.
- Chart Adv.
- Vacuum Pump
- Valve B
- Valve A

Operational Status (Indicator Lights

- Component 1
- Component 2 (can be used for second component)
- Heater 1
- Heater 2
- Sample
- Flame Out

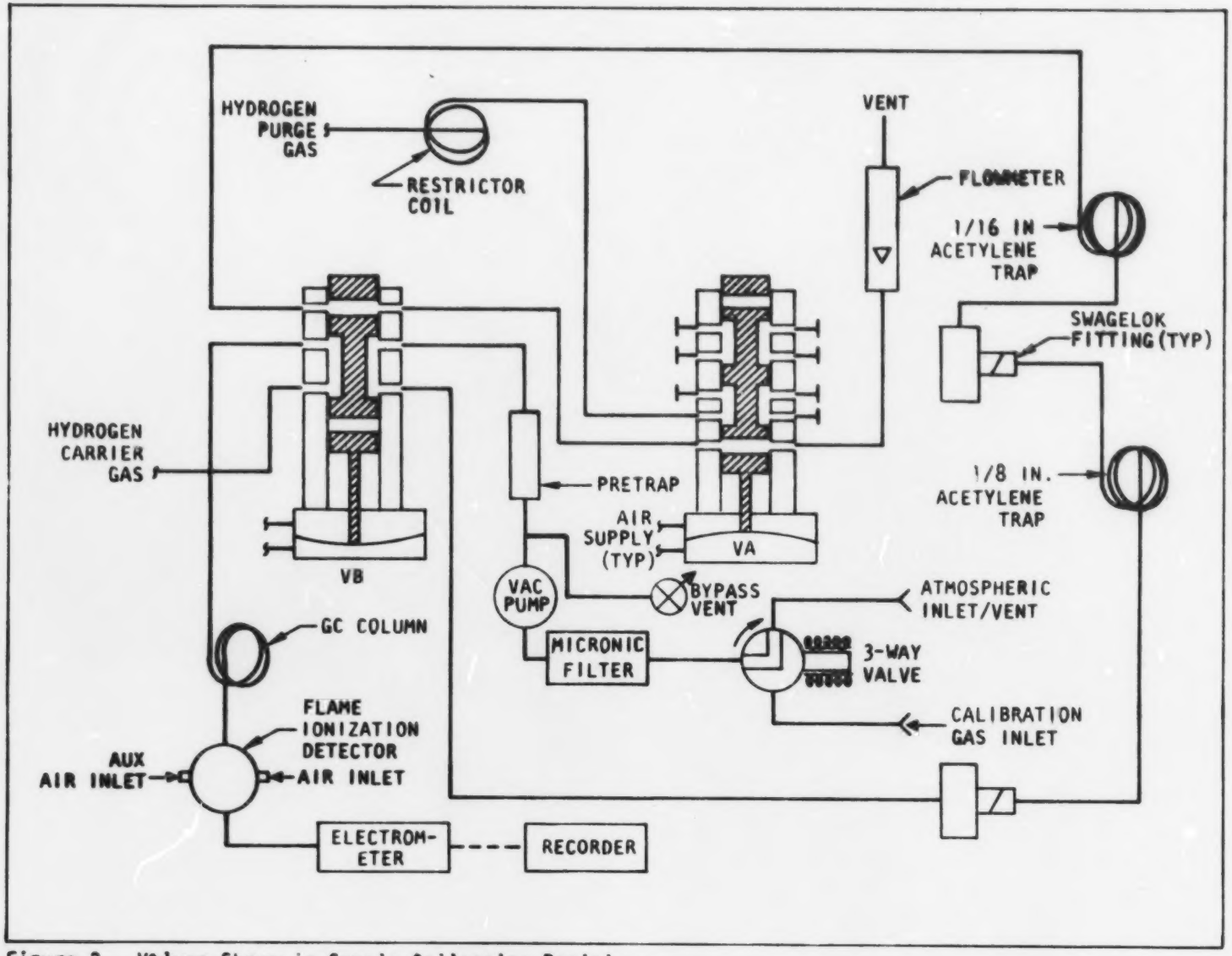

Figure 2. Valves Shown in Sample Collection Position

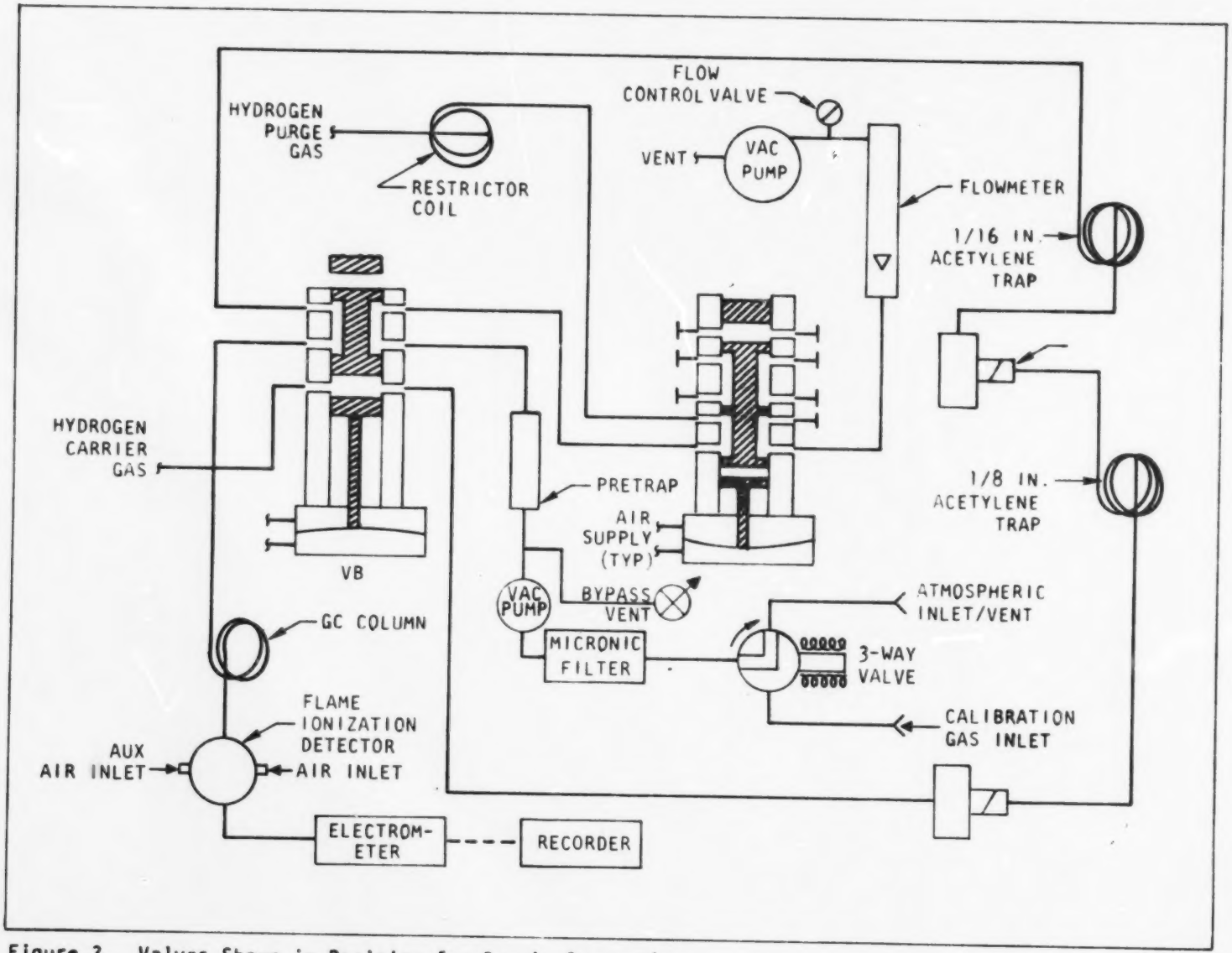

Figure 3. Valves Shown in Position for Sample Separation

function of the pretrap is to remove water and carbon dioxide from the sample. The pretrap consists of 8 inches of anhydrous magnesium perchlorate plus 3 inches of lithium hydroxide packed in a length of 1/4-inch OD Teflon tubing.

The sample gas flow rate and instrument timing is adjusted so that a total of one liter of atmospheric sample is drawn through the trap. When the one-liter sample has been passed through the traps, the instrument automatically stops the sample flow and proceeds with the sample analysis.

While the sample is being collected in the trap, hydrogen carrier gas flows through valve V-B, the gas chromatograph column, and the detector. Gas flow through the column and detector is maintained at all times to keep both the detector and the column equilibrated.

Sample Analysis - After the acetylene has been collected in the trap, the vacuum pump is turned off and valves V-A and V-B are driven sequentially to the position shown in Figure 3 (both valves on) to permit injection separation and measurement of the compounds collected. To accomplish this, hydrogen carrier gas is routed through V-B, through the heated sample traps, into the GC column, and from there to the flame ionization detector. Valve V-A is turned on 20 seconds ahead of V-B. This purges the air from the traps and fills them with hydrogen.

While the sample is being analyzed in the GC column, hydrogen purge gas is passed through a restrictor, V-A, and V-B to backflush the pretrap. Back-flushing removes some of the heavy organic compounds that have accumulated on the pretrap.

In the position shown in Figure 3, Valve V-A also blocks the flowmeter and vacuum pump section of the sample inlet line.

Acetylene Trap Design Objectives - The objective of trap design is to assure that there is a complete trap of subparts per billion of acetylene out of at least one liter of ambient air. Trapping is done at ambient temperatures. The trapping is as selective for acetylene as possible. The trapped sample of acetylene is eluted completely from the trap into a gas volume of one milliliter or less. The traps have a storage life of at least 3 weeks. They are also required to store trapped samples of acetylene for at least 3 weeks without degradation. The latter two requirements must be further verified with field testing of the system (see Task 11 of the Specification Conformance, Section V).

Trap packing material - After some experimentation, Beckman found that acetylene could be trapped quantitatively on a 1-1/2-inch section of 1/8-inch tubing packed with Molecular Sieve 5A which had been treated

with silane*. The acetylene present in one liter of a 50-ppm mixture of acetylene in air was held on this 1/8-inch trap. Heating this trap to 225° to 250°C desorbed 100 percent of the acetylene. Less than 0.5 ppb of acetylene in air was readily detectable by this technique.

Trap construction - Two traps are used in the instrument. The first is a 1/8-inch OD (0.069" ID) tube containing 1-1/2 inches of silane-treated Molecular Sieve 5A. This trap collects all the acetylene from one liter of air. When collection is complete, the first trap is heated rapidly to elute its contents into a second trap of 1/16-inch OD (0.042° ID) tubing also containing 1-1/2 inches of silane-treated Molecular Sieve 5A. Both traps are made of 316 stainless-steel tubing. The 1/16-inch trap is then rapidly heated to elute the acetylene into the GC column for separation from other compounds held by the trap. The location of the 1/8- and 1/16-inch traps outside the instrument is shown in Figure 4. Results of experiments showed that the acetylene peak width was 15 to 30 s wide at one-tenth height when eluting from the 1/8-inch trap, and 10 seconds wide when eluting from the 1/16-inch trap.

The 1/16-inch trap is a permanent part of the instrument. The Remote Sampler uses only the 1/8-inch OD traps to collect acetylene.

Temperature control of the traps - Temperature profiles for the two trap heaters were studied to determine the optimum conditions for elution of acetylene from the traps. The conclusions from these tests are that the traps should be heated to >200°C in less than 15 seconds, and the traps should be held at no greater than 300°C for extended periods of time.

Figure 5 shows how the heater windings are applied to the trap tubing to meet these requirements. Initial tests, runs using a constant-voltage power supply, showed that both of the above requirements could not be met; therefore, the trap heater temperature controller was designed, built, and installed into the system, thereby providing the desired degree of control. A schematic of this temperature controller is shown in Figure 6.

Experimental measurements showed that acetylene can be collected quantitatively at up to 120°F with no loss of acetylene. The sample used for these tests was one liter of 50-ppm acetylene. These results show that no cooling of the trap during sample collection is required.

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*Molecular Sieve 5A was silane-treated by immersing it in dichlorodimethyl silane. The excess silane was drained off and the Molecular Sieve was dried. This silane is available from Matheson, Coleman, and Bell.

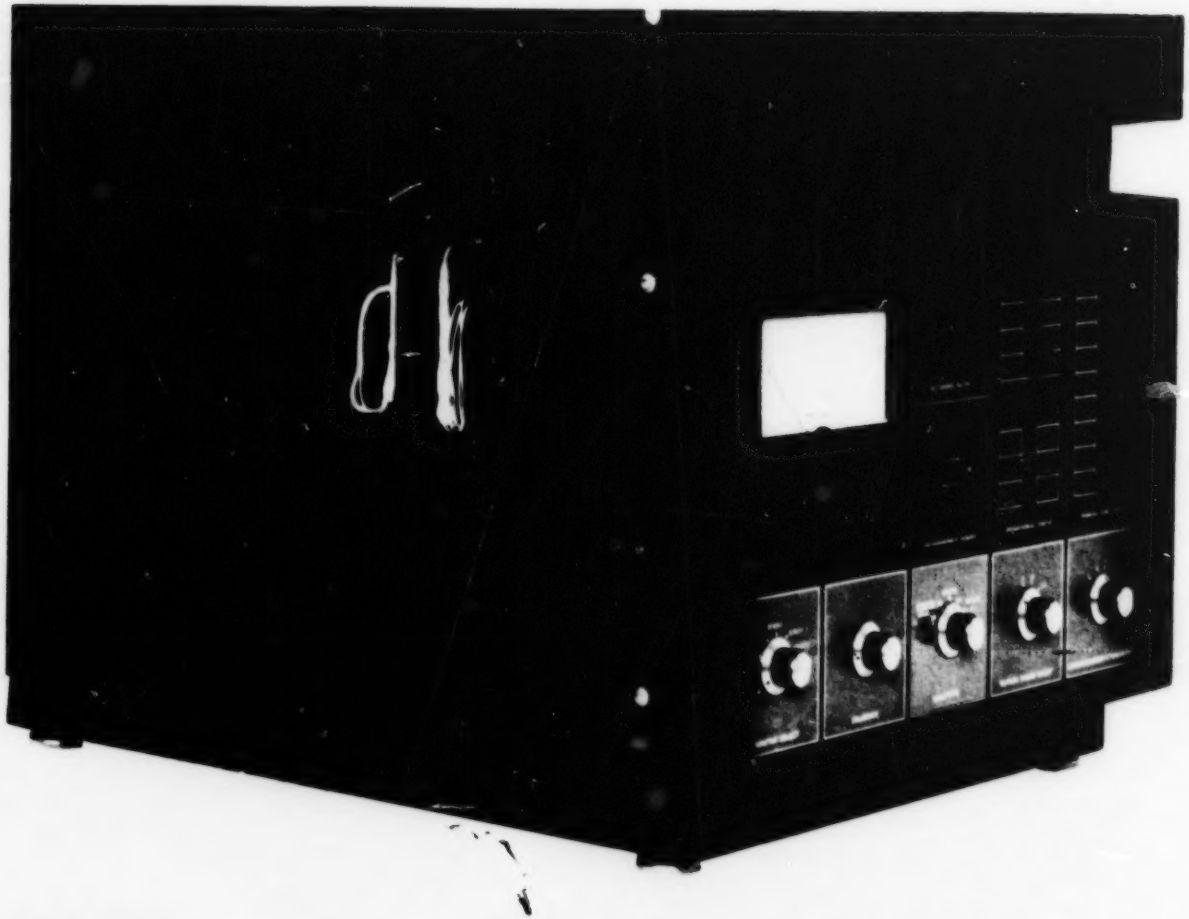
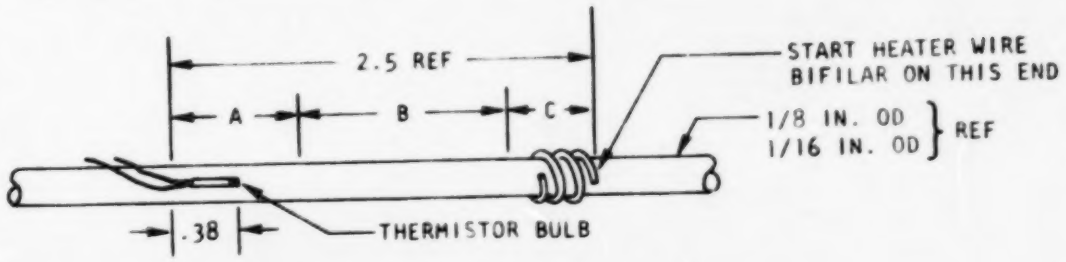


Figure 4. Acetylene Analyzer Showing the Two Traps Mounted on the Outside of the Instrument Case



AREA	1/8 IN. OD		1/16 IN. OD	
	LENGTH ~IN.		LENGTH ~IN.	
	TUBE	HEATER	TUBE	HEATER
A	.63	11.0	.75	22.0
B	1.37	11.5	1.25	35.0
C	.50	7.5	.50	15.0
		30.0		72.0

NOTES

1. WIND HEATER WIRE UNIFORMLY WITHIN EACH DESIGNATED AREA
2. LENGTH SHOWN SHOULD BE HALVED SINCE HEATER WIRE IS BIFILAR WOUND

Figure 5. Heater Winding Instructions for the Acetylene Traps

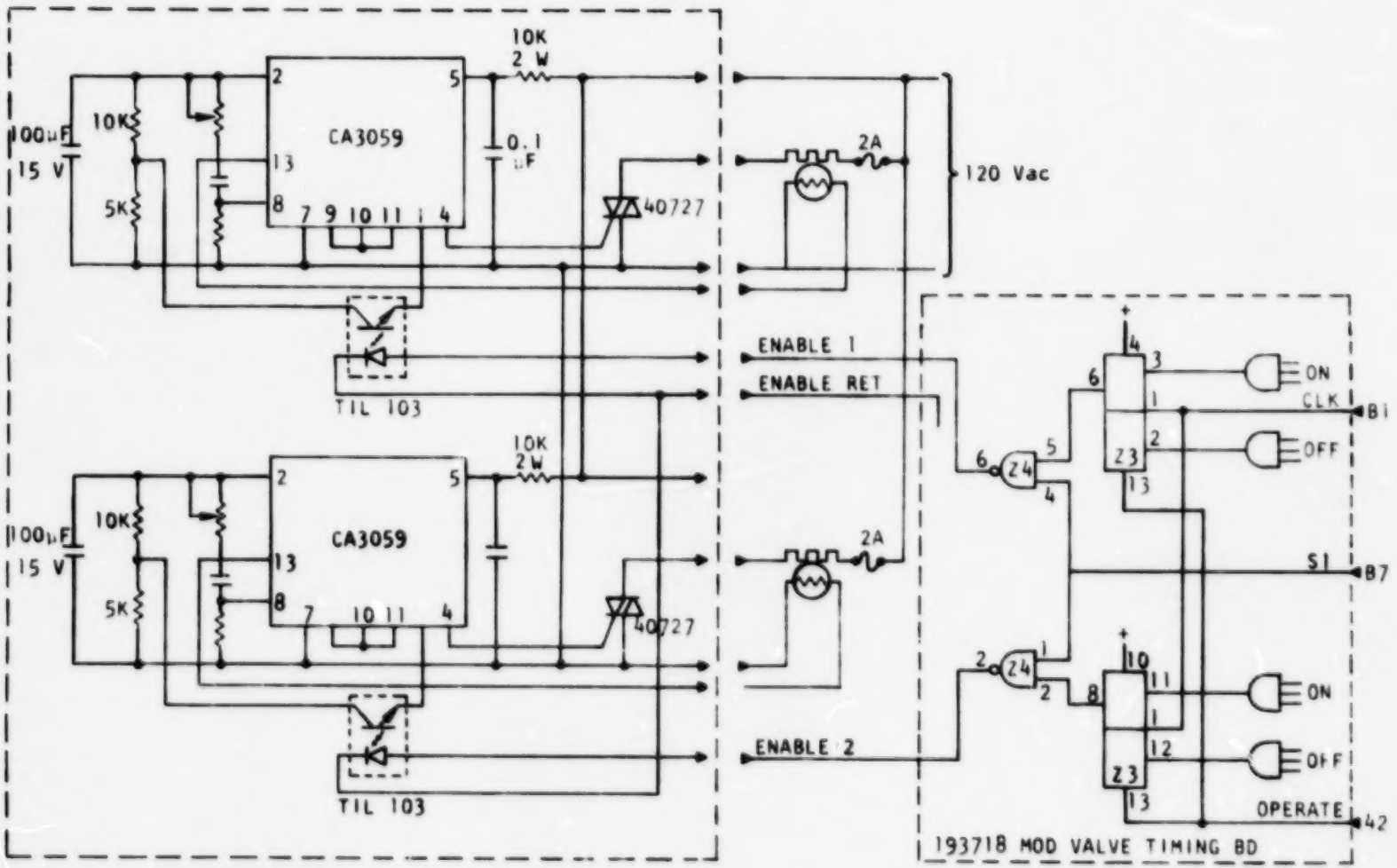


Figure 6. Trap Heater Temperature Controller Schematic

Instrument Operating Sequence

The instrument operating sequence in the automatic mode is shown in Table 2. This table lists the specific action taken at each time interval in the analysis cycle. What occurs in each portion of the analysis cycle is listed in the table under PURPOSE.

Before commencing operation in the automatic mode, the following procedure must be followed:

1. Adjust sample flow rate so that one liter of sample passes through the sample trap in 392 seconds.
2. Allow oven to come to operating temperature of 130°C.
3. Adjust hydrogen and air flow rates.
4. Ignite and stabilize hydrogen flame and turn on instrument electronics.

The operating sequence shown in Table 2 completes one complete analysis in less than 20 minutes (1 200 s) as specified.

In the sequence shown, the recorder is turned on to record the entire chromatogram. This mode was used in developmental testing at Beckman. If desired, the recorder can be easily programmed to record only the acetylene peak. This will significantly reduce the bulk of the data obtained.

Data taken with an instrument automatically measuring acetylene are shown in Figure 7. These are two 24-hour continuous runs. Manual calibrations were made approximately every 12 hours. The measurements were made at the Beckman plant in Anaheim, California, located in an urban area about one mile from the junction of two major freeways. Note that the acetylene concentration shows peaks in the morning and evening "rush hours" on Wednesday. These peaks are absent from the Sunday data.

REMOTE SAMPLER

The specification requires the construction of a device for collecting acetylene from the atmosphere for analysis at a later date. The Remote Sampler, developed to satisfy this requirement, is shown schematically in Figures 8 and 9.

The Remote Sampler contains three 1/8-inch traps identical to those used in the automatic analyzer. Samples of ambient air are drawn through each of these traps in turn. The operating sequence is controlled by a 24-hour cam timer. The samples are drawn into the traps through a particulate filter and a pretrap which are identical to those used in the automatic analyzer. The sample flow rate through the traps can be adjusted in the field by using a flowmeter with a flow control valve.

Table 2. Instrument Operating Sequence in the Automatic Mode

FUNCTION	TIME (s)	PURPOSE
Vacuum Pump ON	002	
Auto Zero ON	382	
Auto Zero OFF	388	
Vacuum Pump OFF	394	
Valve V-A ON	402	
Recorder ON	402	
Valve V-B ON	422	
Trap Heater 1/8 in. ON	422	
Trap Heater 1/16 in. ON	444	
Valve V-B OFF	460	
Acetylene Peak Elution	636-684	
Trap Heater 1/8 in. OFF	744	
Trap Heater 1/16 in. OFF	750	
Valve V-A OFF	1180	
Recorder OFF	1182	
Recycle through next analysis (20-min. cycle)		
Alternatively--hold for 10 minutes and recycle (30-min. cycle)		

Collect one liter Acetylene from air

Back out detector background current

Purge air from traps and fill with hydrogen

Record chromatogram

Set Valve V-B to ON

Route trap effluent to GC column

Trap heated. Elutes compounds from 1/8 in. trap to 1/16 in. trap

1/16 in. trap heated. Elutes compound from 1/16 in. trap to column

Trap located and back-flushed with hydrogen to vent.

Trap hedted and back-flushed with with hydrogen to vent

Separate trap effluents on GC column

Reset Valve A to sample intake position

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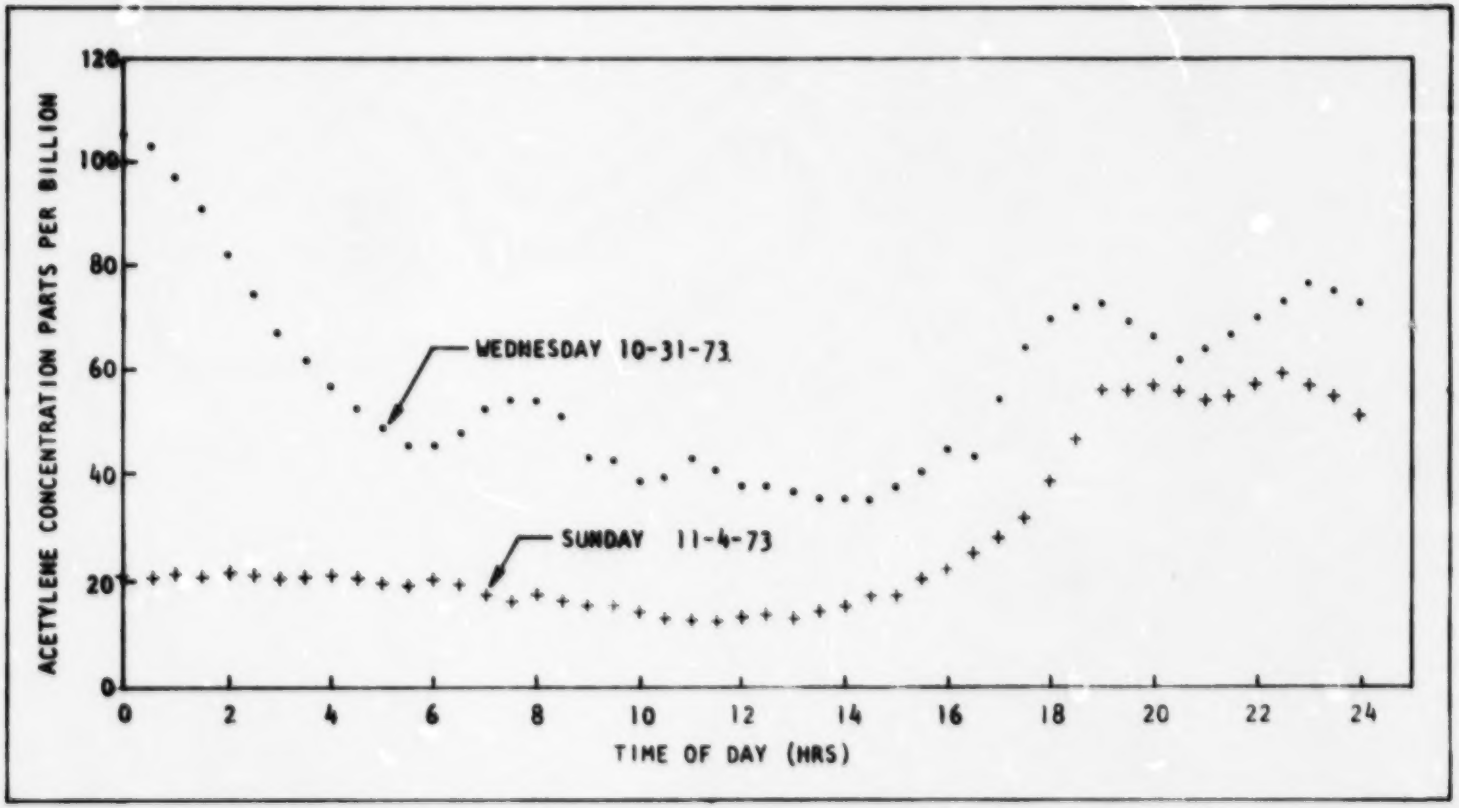

Figure 7. Acetylene Concentrations Measured With the Instrument Operating in Automatic Mode for a Three-Day Continuous Run

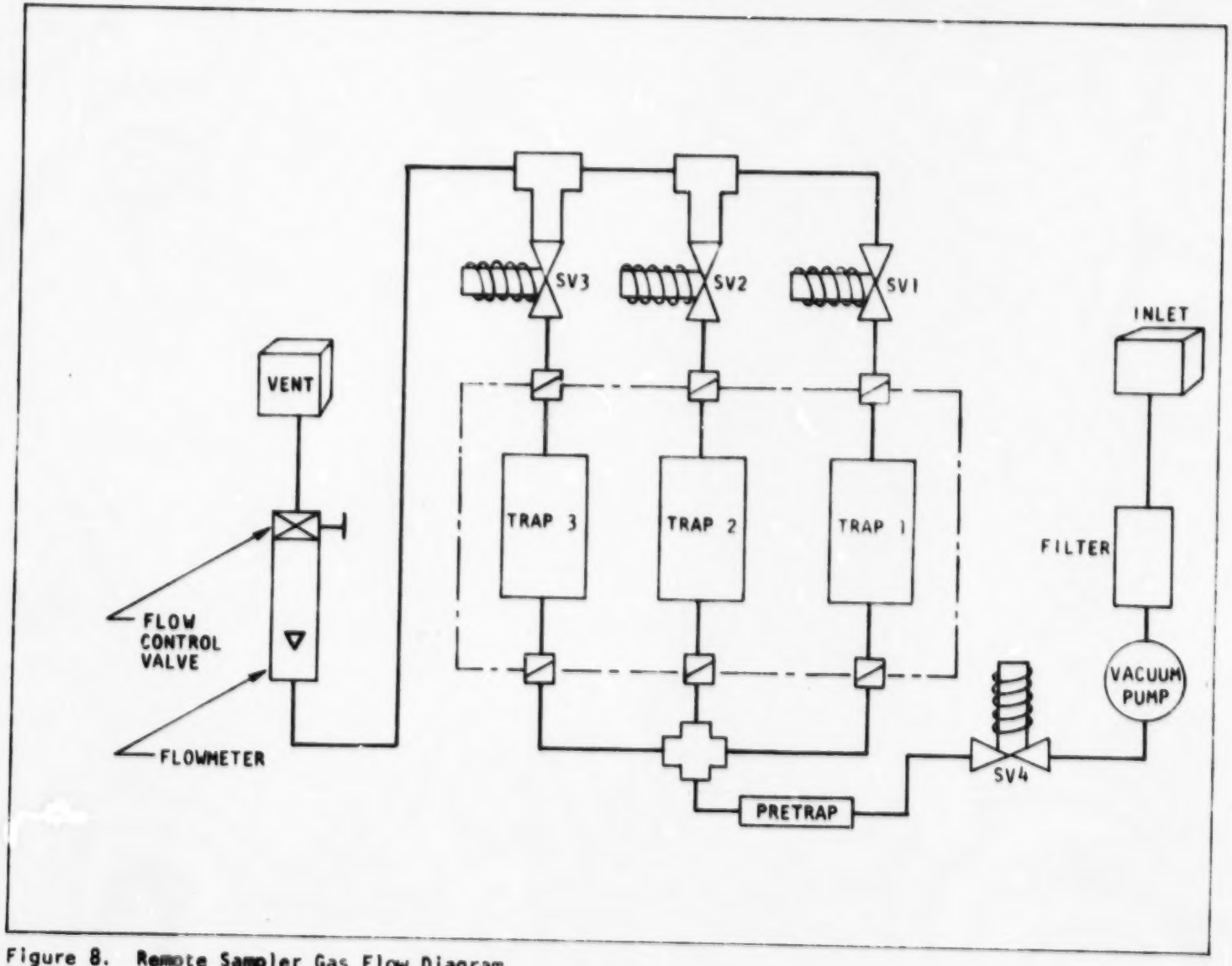

Figure 8. Remote Sampler Gas Flow Diagram

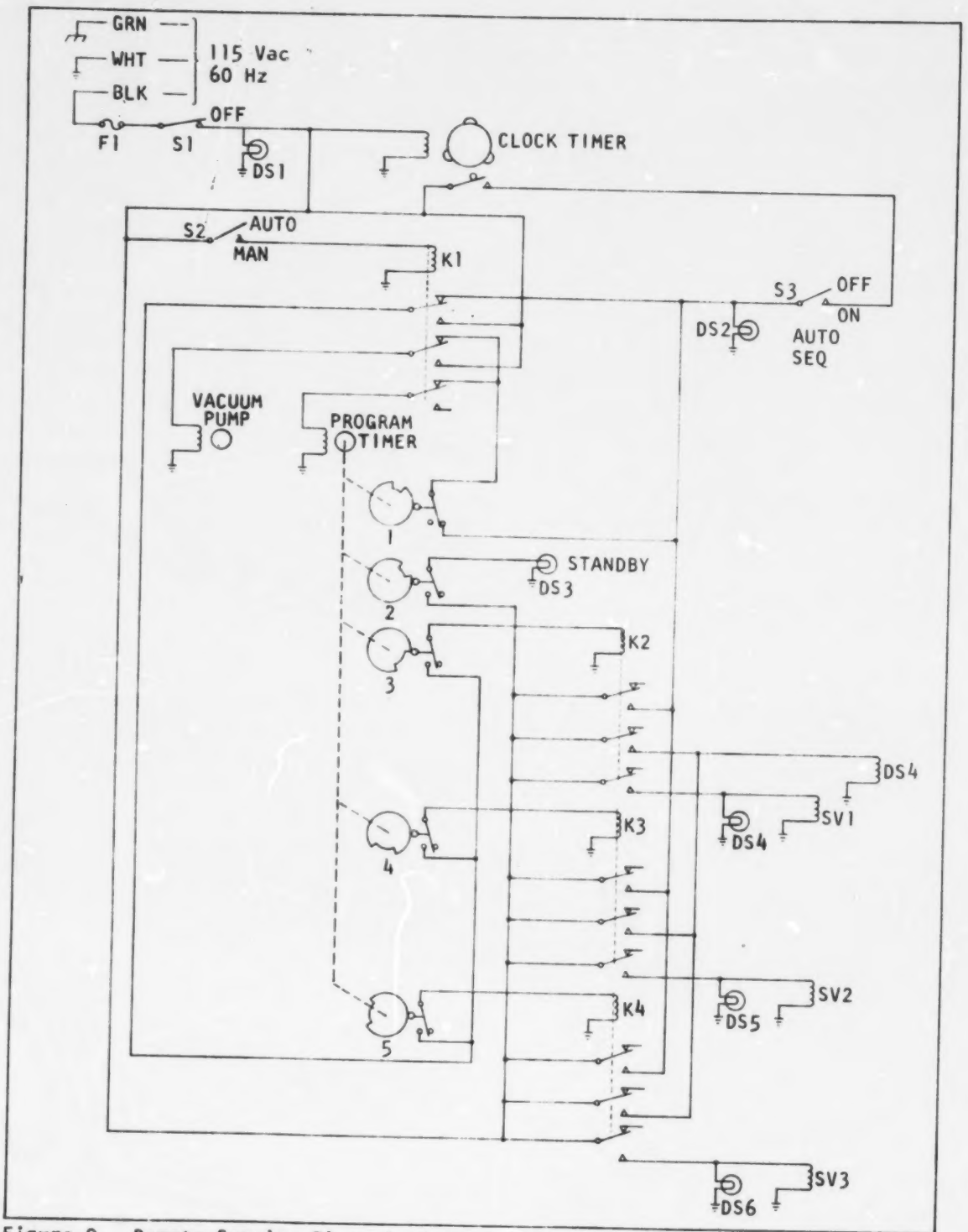

Figure 9. Remote Sampler Electrical Schematic

The sample is drawn through the system with a vacuum pump included in the Remote Sampler case. The gas flow is routed through any one of three traps by solenoid valves. The valves are activated only when a sample is being taken so that each trap is protected from contamination when it is not in use.

The Remote Sampler is powered by 115 Vac, 60 Hz. It is built into a sealed metal case that protects the entire sampler from the weather.

Gas Chromatograph Column

The gas chromatograph column is prepared by packing 3 feet of 1/8-inch OD (0.080" ID) stainless steel tubing with F-1 Alumina* (42-60 mesh). The column is conditioned overnight at 225°C with hydrogen carrier gas flowing.

The column is operated at 130°C with a hydrogen carrier gas flow rate of about 25 ml/min. The flow rate is adjusted so that normal pentane elutes in about 12 minutes.

The column must be highly activated to separate acetylene from normal butane. Water and, possibly, carbon dioxide rapidly deactivate the alumina and cause the column performance to deteriorate. This effect was avoided by putting on an anhydrous magnesium perchlorate plus lithium hydroxide pretrap ahead of the acetylene trap in the instrument.

Figure 10 is a chromatogram obtained with the column described above. The sample was collected on the instrument trap and eluted into the column in the automatic mode. Each peak in this chromatogram is equivalent to one-part-per-billion of the component in a one-liter sample.

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*F-1 Alumina is available from Applied Science Laboratories, Inglewood, CA

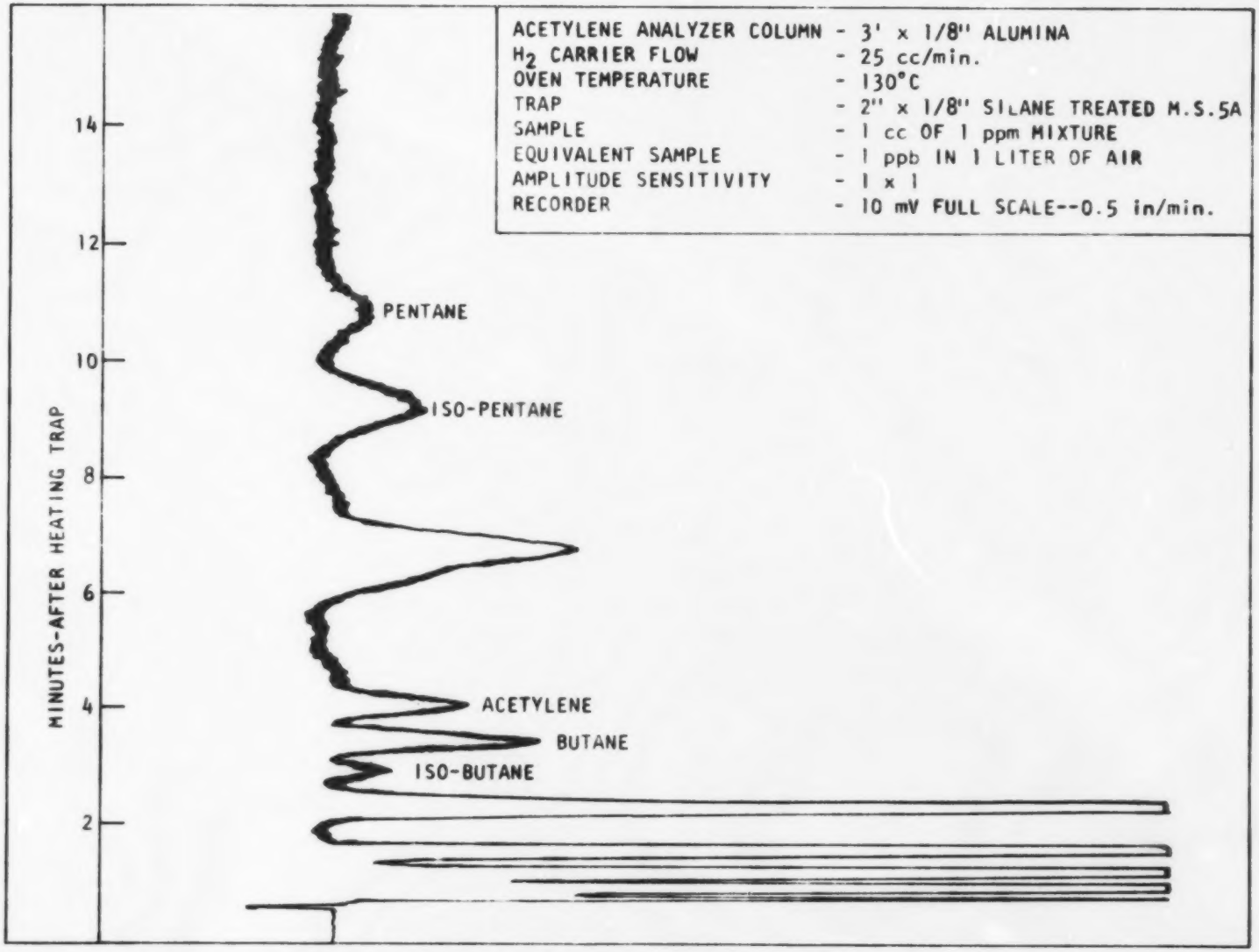

Figure 10. Chromatogram Obtained with the Automatic Acetylene Analyzer

SECTION V

SPECIFICATION CONFORMANCE

This section gives the paragraph from the Work Statement, Scope of Work, and states for each paragraph how the requirements of that particular section were met.

The approach suggested is one using an automated concentration technique to trap the acetylene species in approximately one liter of air, followed by injection of the trapped contents into a gas chromatographic column for separation. The method of detection is the flame ionization detector. The entire system is to be operated by electrically operated pneumatic valves, controlled by adjustable timers. The entire procedure of trapping, injection, and gas chromatographic analysis should be capable of repetition every 20 to 30 minutes.

> The instrument described in Section 3 follows the EPA suggested approach without exception.

Task 1. Construct the acetylene analyzers. The units should be compact and capable of easily transporting from one sampling station to the next.

> Analyzers have been constructed that are easily transportable. They occupy a 17 x 40 x 20-inch envelope and weigh 250 lb when packed for shipment.

Task 2. The amplifier used in the construction of this analyzer should have a sensitivity of 5×10^{-12} ampere full-scale maximum, and a background baseline noise lower than 10^{-13} amps.

> The amplifier in the instrument exceeds the sensitivity requirements. Its sensitivity is 2×10^{-12} amps full-scale maximum with a noise lower than 10^{-13} amps.

Task 3. Choose gas chromatographic substrate and trapping material.

> The gas chromatographic substrate is discussed in Section IV. The trapping material is also described in Section IV.

Task 4. Set up the gas chromatographic and trapping procedures in automatic modes.

> The automatic timing sequence is given in Section IV. This sequence includes both the trapping and gas chromatographic operations.

Task 5. Determine trapping capacity and efficiency for the acetylene species.

The trap was shown to hold all of a one-liter, 50-ppm acetylene sample in air. One liter of a 50-ppb sample was completely retained on the trap after 3 liters of air were passed through the trap.

Task 6. Evaluate the temperature required to desorb acetylene from the trap with 100-percent efficiency.

During the analysis cycle, 100 percent of the acetylene was shown to be desorbed from the trap. Over 90 percent of the acetylene is desorbed into the GC column; the remainder is backflushed. The trap is heated to between 225° and 250°C to desorb the acetylene.

Task 7. Determine trapping efficiency and desorption characteristics of iso and normal butane, iso and normal pentane.

The trap may be thought of as a short GC column. Isobutane elutes through the trap in one or two minutes with the normal sample flow rate. Isopentane elutes in four minutes under the same conditions. Both normal butane and normal pentane are retained on the trap for the time required to pass the usual one-liter gas sample. Therefore, these two normal hydrocarbons will be trapped quantitatively and the amount in the air can be measured since these hydrocarbons are separated cleanly in the GC column. These two iso compounds will appear in the chromatogram but the quantity present in the air cannot be accurately measured because of large losses from the trap.

Task 8. Determine the feasibility of employing a thermoelectric cooler to improve the trapping efficiency of acetylene.

Since the trapping efficiency of acetylene is 100 percent at ambient temperatures for concentrations in the range of interest, it is not necessary to use any form of trap cooling to improve trapping efficiency.

Task 9. A device should be constructed for collection of acetylene for subsequent analysis at a later date, thus making it possible to collect many samples at various sites.

The Remote Sampler, described in Section IV, was designed and built to satisfy this requirement.

Task 10. Check the effect of humidity on trapping efficiency.

Humidity has no effect on trapping efficiency since pretrap packed with magnesium perchlorate, which removes essentially all the water from the sample gas, is employed.

Task 11. The stability of the trapping material in the solid device described in Task 9 should be tested—both before and after collection of the acetylene. The trapping material should have a shelf life or stability of at least three weeks prior to sample collection and three weeks after collection.

A series of traps with sample was placed in storage. Performance of these traps could not be determined because of initial miscalibration of the instrument.

Task 12. Determine minimum detectability of the analyzer to the acetylene species.

The minimum detectability of the analyzer was found to be less than 0.5 ppb.

Task 13. Evaluate the system in field experiments.

Beckman supported EPA in a series of field tests of this instrument. These tests were conducted in March 1974 at Research Triangle Park. The results of these tests showed that this instrument functioned (detection limit and stability) as required by the contract.

Task 14. Report the results of all requested experiments in tables, graphs, and verbal descriptions.

The results of all requested experiments have been reported in this Final Report, the monthly reports, or verbally to the EPA Technical Officer.

Task 15. Furnish the project officer with two finished instruments with the desired characteristics, complete details of the necessary capabilities, and a solid collection device that can be used to collect acetylene at subparts per billion and stored for at least three weeks, and from which the acetylene can be desorbed quantitatively into a gas chromatograph for precision analysis.

The first instrument was shipped to EPA February 18, 1974. The second instrument was delivered after field experiments on the first instrument had been completed. Complete details of the instrument capabilities are given in this report and in the Instruction Manual supplied with the instrument. Each of the two automatic instruments delivered contains a solid collection device having the required characteristics.